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Journal of Sedimentary Petrology

A Publication of the Society of Economic Paleontologists and Mineralogists
a Division of
The American Association of Petroleum Geologists

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Articles submitted for publication to the *Journal of Sedimentary Petrology* should have a prefatory abstract and sufficient postage should be sent by the author to prepay the return of the manuscript as it has been found that many articles require more or less revision before publication.

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DECEMBER, 1933

THE PETROLOGY OF THE MARSHALL FORMATION OF MICHIGAN

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University of Michigan

ABSTRACT

Study of the petrology of the Mississippian Marshall formation of Michigan indicates that the materials of the formation were chiefly derived from the Laurentian and Huronian terranes or from sedimentary rocks originally derived from these terranes and that the distribution areas were closer to the sites of deposition of the western parts of the state than to the eastern.

Attention has been focused on the Mississippian Marshall formation throughout the history of the state. In the early days it was important as a source of brine, building stone and grindstones, but today the chief interest lies in its use as a key horizon to which test wells are drilled to obtain information relating to structure. It is the most widespread of the near-surface deposits and covers almost the entire Lower Peninsula with the exception of two tiers of counties in the northern part of the state, and about two tiers in the southern part (fig. 1). Outcrops are infrequent due to the deep covering of glacial drift.

LITHOLOGY

The formation is composed of the lower Marshall, and the upper Marshall or Napoleon. The lower Marshall consists of sands and shales with occasional limy layers and, in eastern Michigan a conglomerate containing small pebbles of peanut size often marks the change from the underlying Coldwater shales. There are many layers of red shaly sandstone which usually have a high lime content. In the center of the Michigan basin this "red rock" horizon seems to be fairly consistent in denoting the change from lower to upper Marshall. In the eastern part of the state, the position of the red layers seems erratic, but they may mark the change from lower Marshall to Napoleon. In some instances this change is indicated by a concentration of muscovite, or a conglomerate consisting of peanut size pebbles, and the "red rock" is then found near the bottom of the lower Marshall.

On the northwestern side of the state, in localities where deep drilling has yielded the information, the two members of the Marshall are practically indistinguishable and the red color and shaly characteristics prevail throughout the formation.

In southwestern Michigan the Marshall formation is thin and the red color is rare.

The lower Marshall has a high iron content and contains much muscovite. The cement is usually lime.

The upper Marshall is a medium-to coarse-grained porous sandstone which in the exposures shows cross-bedding, and a conglomerate is usually found

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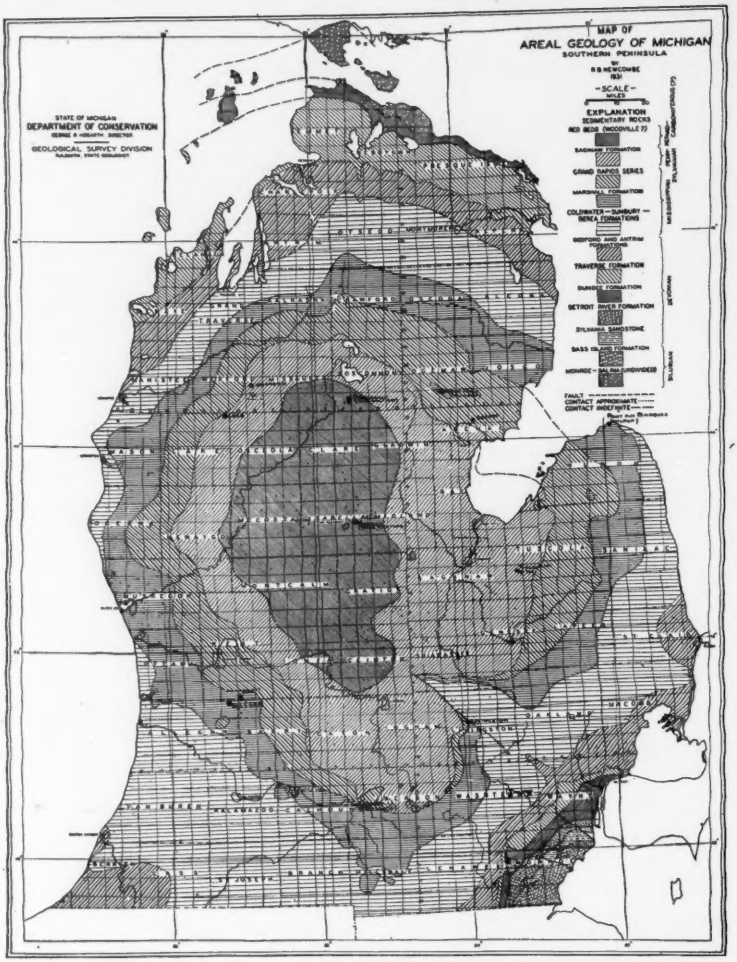

FIG. 1. Geologic map of Michigan showing location of outcrops and wells from which samples were used.

at its base. The color is buff to greenish and is spotted with white or brown resulting from alteration of some constituents. The cementing material is siliceous.

The change from the upper Marshall to the overlying Michigan formation is often indicated by a thin green shale. Near the bottom of the Michigan formation a sandstone of irregular

geographical distribution has been recently discovered. Its lithologic similarity to the Napoleon sandstone led to the conclusion that the Michigan sandstone was derived from the erosion of islands of upper Marshall that projected above the Michigan sea (Newcombe, 1931, p. 84).

The structures of the Marshall sandstone coincide in a broad way with those of the other Paleozoic sediments of the region. The Michigan synclinal basin was early subjected to warping which formed synclines and anticlines within the major syncline. All the Paleozoic deposits, so far as known, follow the irregularities of these original control lines. This condition makes it possible to use the younger formations as a guide to the structures existing in the older beds.

Many small variations found at the top of the upper Marshall are topographic rather than structural, resulting from a period of erosion at the close of Marshall time. Thomas (1930, p. 498) notes a pronounced unconformity between the upper Marshall and the underlying beds on the western side of the state. The Marshall on the eastern side of the state, he says, is conformable to the lower formations, and it is in this region that the Marshall is particularly important as a key horizon. On the western side the usual thickness of the formation is approximately 200 feet, whereas in Huron County 450 to 400 feet are common.

PETROGRAPHY OF THE SEDIMENTS OF THE MARSHALL FORMATION

Both the Pure Oil Company and the State Geological Survey kindly provided samples from various locations for a petrographic study of the Marshall formation. The samples were given with the understanding that the exact locations be confidential and thus it was not possible to work out a detailed correlation of particular horizons. An attempt was made to obtain a general knowledge of the depositional environment of Marshall sediments, and, based upon a study of the heavy minerals, of the source from which the material of the sandstone was derived.

The samples from Arenac, Tuscola, Livingston, Kent and Allegan Counties (see fig. 1) were taken from core test wells drilled with a light hollow-rod drilling machine. Samples from wildcat wells in Wexford and Lake Counties were also examined. In addition to well cuttings, outcrops from the bed of Rifle River, section 9, Mills Township, Ogemaw County, and from Point aux Barques, Port Austin Township, Huron County were studied. Two thin sections were obtained from the Grayling Development Company's diamond drill cores in Roscommon County, one from a diamond drill core of an Isabella County well, and another from an outcrop in Sanilac County.

Six outcrop samples, three diamond drill cores, and 269 well cuttings were examined during this study. The petrographic determinations were compiled from four thin sections and 279 slides of detrital material.

LABORATORY PROCEDURE

Each bag of the well cuttings is presumably an average sample of 5 feet of the rock section. Fifty grams of each sample were weighed and allowed

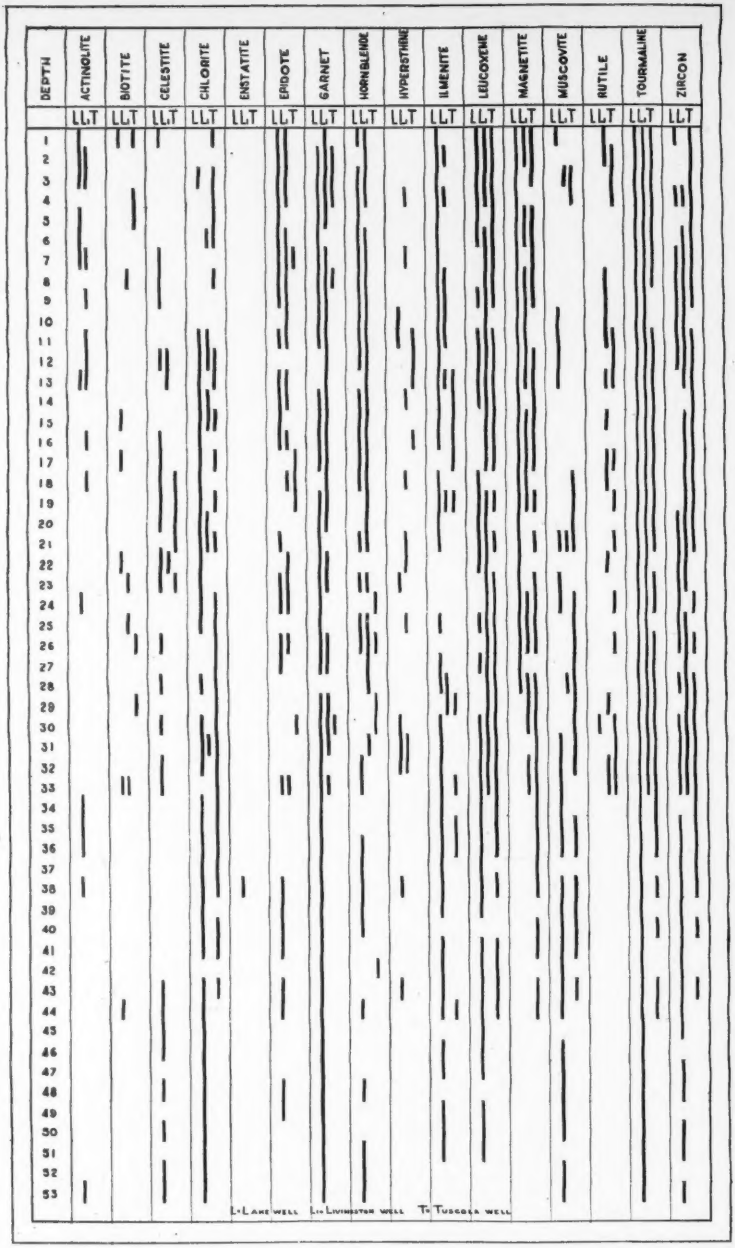

FIG. 2. Graph showing comparative mineral distribution for the Tuscola (T), Livingston (LI) and Lake (L) County wells.

Figures on the vertical scale represent five foot samples in descending order from number 1—the top of the Marshall.

to stand in a 30 per cent HCl acid bath for 12 to 24 hours, depending on the amount of ferruginous matter present, then washed and dried, and the total loss calculated. Particles less than 0.05 mm. in diameter were lost during the washing process.

For use in the heavy liquid separation, the insoluble residue was screened and the fraction below 0.25 mm. in diameter saved. The size of the sample used for this separation depended on the amount of material available, but wherever possible 15 to 20 grams were taken. Bromoform, which averaged 2.82 in specific gravity, was used as the heavy liquid. Separations were made according to Milner's method (1929, pp. 43-54) and alcohol was used for the washing medium. In general, the residues were so completely flooded with pyrite that other constituents were obscured and to remove the pyrite the grains were submitted to a 30 per cent HNO_3 acid bath for about 8 to 10 hours, and in most instances re-separations were made to eliminate quartz that had been carried down in association with pyrite.

A DESCRIPTION OF THE MARSHALL MINERAL SUITE

The mineral suite of the Marshall formation is neither abundant nor very diversified. Of the light minerals quartz is the most plentiful, forming the bulk of a sample. Minor quantities of feldspar along with its kaolin and sericitic alteration products are also present. Pyrite, magnetite, ilmenite, and leucoxene are the most abundant accessory minerals. After the removal of the opaque species tourmaline remains as the most persistent and common mineral of the eastern region with zircon almost equally frequent and geographically as widespread. Garnet exceeds tourmaline in the western suite of minerals. Actinolite, biotite, chlorite, epidote, hornblende, hypersthene, muscovite, and rutile represent the minor constituents among the high specific gravity residues. Celestite and dolomite are occasionally present.

The western residues are more varied and abundant than the eastern and contain garnet, which because of its stability indicates that the differences between the two suites are inherent, and not the result of varying stages of alteration. Pink zircon, hornblende, epidote, actinolite, and some biotite are also characteristic.

The eastern assemblage is typical of a reworked sediment, and contains only such resistant minerals as tourmaline and zircon, and the alteration products of less stable species. The scarcity of garnet and the absence of pink zircon are conspicuous.

COMPARISON OF THE EASTERN AND WESTERN ASSEMBLAGES

Lake and Tuscola Wells.—The Lake and Tuscola wells were chosen for the comparison because they are representative of the general variety of minerals found in their respective localities, and they constitute the most complete sections available for examination from either side of the state.

The light minerals of the wells are similar. Both contain small amounts of microcline, orthoclase, altered plagioclase, and sometimes muscovite and chlorite. The greatest percentage is

made up of angular to subangular quartz grains, most of which contain rutile arranged in fine parallel lines. The heavy residues are flooded with pyrite. Magnetite is present in only slightly smaller quantities.

The Lake County samples show the most striking difference from those of Tuscola County in the frequency and persistence with which garnet appears in the Lake County residues. Pink, orange-red, and colorless garnets occur in that general order of frequency for the entire section, but any one of these three colors may dominate in a single slide. In the eastern well garnets are present in but four of the 44 slides, and they are either pink or colorless, never red. Two is the largest number of garnets ever found in a Tuscola slide, whereas 10 to 15 may occur in one from Lake County.

Unstable minerals such as hornblende and actinolite appear frequently in the Lake County samples without much sign of incipient alteration. Actinolite does not occur in the Tuscola residues at all, and hornblende is rare. That the latter has been replaced in most instances by chlorite is known from observing grains in which the process is incomplete. Epidote and hypersthene are more persistent and abundant in the western well than in the eastern region.

Differences in the ilmenite-leucoxene

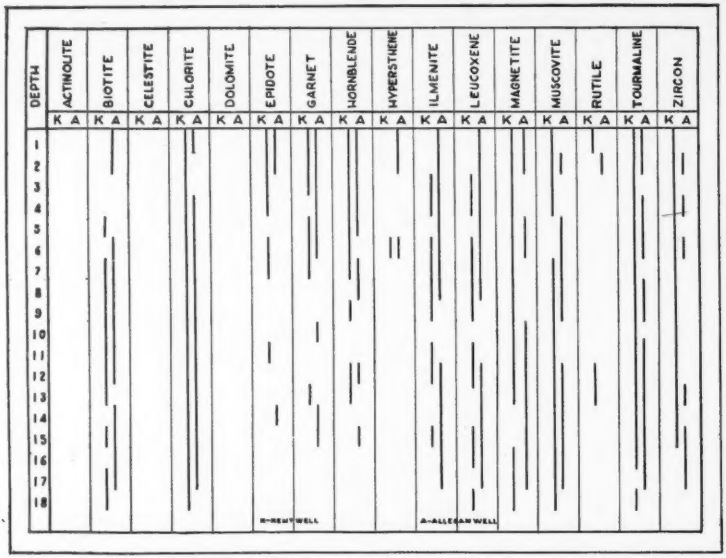

FIG. 3. Graph showing comparative mineral distribution for the Kent (K) and Allegan (A) County wells.

Figures on the vertical scale represent five foot samples in descending order from number 1—the top of the Marshall.

ratio are not so apparent on the graph as they are on the slides. However, in the Tuscola well leucoxene is persistent whereas ilmenite does not appear until a depth of 55 feet below the top of the Marshall (see fig. 2). Leucoxene is recorded as "on ilmenite," or simply as "leucoxene," showing it to be the more important of the two constituents. In the Lake County sands, on the other hand, ilmenite is very persistent and is described as "ilmenite—with little leucoxene alteration," or "Ilmenite flooding—little leucoxene," indicating that ilmenite is the dominant form. Furthermore, many ilmenite grains of the western residues have fresh unaltered surfaces, an occurrence which has never been observed in the Tuscola samples.

Thus we see that the Lake and Tuscola residues have distinct heavy mineral assemblages, which may be summarized as follows:

Lake County: The abundance of fresh ilmenite, hornblende, actinolite, and epidote, together with the presence of garnet and pink zircon characterize this suite of minerals.

Tuscola County: Abundant products of alteration such as leucoxene and chlorite, rarity of garnet, and total absence of pink zircon and actinolite differentiate this assemblage from the Lake County suite.

Discussion of All Eastern and Western Residues.—No actinolite is present in the samples from the Kent and Allegan wells (fig. 3). Fresh hornblende is common and quite persistent. Epidote is plentiful when it occurs, but is not as well distributed throughout the section as in the Lake County samples. Garnets are common and the orange-red color is a characteristic feature. Ilmenite is present with fresh unaltered surfaces whereas leucoxene is a minor constituent. Pink zircon is frequent.

Garnet and epidote are rare in both of the Tuscola wells, and in the Arenac samples. No actinolite is present; hornblende is infrequent and shows signs of alteration when it does appear. Leucoxene is almost universally present, but ilmenite was not often observed. Neither was pink zircon found.

It is apparent that the eastern residues characteristically show the effects of alteration in the presence of large amounts of leucoxene and minor quantities of ilmenite; by the absence of most of the ferro-magnesian minerals or the advanced stage of weathering in which the hornblende occurs. Orange-red garnets are absent, and the pink to colorless varieties are scarce. Only colorless zircon appears.

The western assemblage is characterized by the presence and abundance of garnet—particularly the orange-red species; by pink zircon; and by the dominance of fresh ilmenite and ferro-magnesian minerals.

RELATION OF LIVINGSTON RESIDUES TO THE EASTERN AND WESTERN ASSEMBLAGES

The Livingston well is located (fig. 1) on the southwestern or downthrown side of the Howell-Owosso fault and the entire thickness of the Marshall was available for study. The suite of minerals is large and the variety of species is as great as in the Lake County well (fig. 2).

The vertical continuity and the

abundance of garnet, particularly the orange-red variety, is very striking. Hornblende, epidote, and actinolite are also common and persistent. The presence of garnet, and the freshness of the unstable minerals in the Livingston suite indicates a close relationship to the western assemblages. From the standpoint of the ilmenite-leucoxene ratio the Livingston residues have a greater resemblance to the eastern samples. Ilmenite is somewhat more abundant than in the eastern suite, but it is not as common as in the typical western residues.

The presence of fresh amphiboles, which are themselves unstable, argues against prolonged transportation as the reason for such an increased percentage of leucoxene. According to Milner (1929, p. 445) the causes of transformation from one titanium mineral to another are not well known but he suggests that the ilmenite form is more common in connection with an iron mineral environment. Therefore in the Lake County samples where iron minerals dominate, ilmenite is also abundant, whereas in the Livingston area where iron minerals are much less important, leucoxene is more frequent.

Another possible explanation is that the position of the Livingston well close to the fault line made these samples subject to alteration by meteoric waters. On the basis of the conditions in the Kent and Allegan wells and on their similarity to the Livingston conditions, however, the first explanation is probably preferable.

Actinolite in the Livingston suite, since it does not appear in the Kent and Allegan residues, presents another problem. The latter wells, however, show only the lower 92 feet of the Marshall section and in the Livingston samples actinolite is not found in the lower 73 feet of Marshall sandstone. It is probable that the lower portion of the Marshall underwent a more rigorous decomposition than was suffered by the younger sediments leading to destruction of the actinolite of the Kent and Allegan wells, and the lower part of the Livingston section.

It is obvious that a close affinity, evident on the basis of the orange-red garnet alone, exists between the Livingston and western assemblages. It seems strange that this should be true when the Livingston and Lake wells are separated by 140 miles, as compared to a distance of 70 miles between the Tuscola and Livingston locations.

On the northeastern side of the fault which has already been mentioned, an anticline separates the Tuscola and Livingston localities. In spite of extensive testing no Marshall outliers have been discovered on the upthrown side of the fault, and it is possible that the Marshall sea never covered this region. If such a barrier did exist, it would prevent a mingling of the eastern and southern drainage systems, and the two sides of the fault would have unrelated suites of minerals as long as differences existed in the source rock from which each was derived.

THE SOURCE OF THE MATERIAL AND THE ENVIRONMENT OF DEPOSITION OF THE MARSHALL FORMATION

Problems of the Distributive Province.—In order to determine the sources of the materials and the con-

ditions under which Marshall sediments were deposited, the distributive province, or provinces must be known. Two major considerations, namely, the rocks contributing to the basin, and the features which controlled the direction of drainage must be understood. For this purpose a short résumé of the geology of the area adjoining Michigan is attempted.

Structures Delimiting the Province.—The Cincinnati arch: To the south of Michigan the Cincinnati arch is the major structural feature. The arch developed in late Ordovician or early Silurian time, and influenced drainage through much of the subsequent Paleozoic.

The Indiana extension of the structure has been described by Malott (1932, p. 128) to have its axis stretching from the crest in Jessamine County, Kentucky, northwestwardly toward Richmond and Logansport, Indiana, descending in this direction until in the vicinity of Logansport the strata rise again along the axis which here trends toward the region of Chicago. Eventually the arch becomes a part of the Wisconsin uplift.

On the northwestern side of the Indiana arch the dips are low and the rocks incline into the Michigan basin.

Wisconsin Uplift: The Paleozoic sediments dip away from the crest toward the west and east according to Martin (1911, p. 87). This uplift passes northward through the central part of Wisconsin.

Pre-Cambrian Highlands: The northern uplands of pre-Cambrian rocks constitute almost the entire Lake Superior district and extend through Canada, past the northern end of Lake Huron toward the eastern coast of North America. A neck of pre-Cambrian rock extends southeast and, crossing the St. Lawrence at the Thousand Islands, divides the Paleozoic rocks into two distinct areas.

Ontario Peninsula: Passing southward from the Lake Superior area into the Ontario Peninsula of Canada, Paleozoic sediments show the presence of rather small dips toward the Michigan synclinal basin, and away from the central pre-Cambrian axis. This southwesterly dip continues to the region lying between Lake Erie and the south end of Lake Huron. From this section a gradual rise of the strata toward the southwest occurs (Malcolm, 1915, p. 45).

Summary of Structural Control.—It is evident from the foregoing that Michigan is surrounded by a set of structures which isolated the basin throughout much of Paleozoic time. In the southern region the Cincinnati anticline has acted as a barrier since late Ordovician: toward the west the Wisconsin arch has resulted in dips toward the Michigan basin: in the north the pre-Cambrian uplands of the Lake Superior region have enclosed the basin in that direction, on the east, dips have been away from the pre-Cambrian mass, which in Ontario resulted in inclination toward the Michigan basin.

Thus drainage has been toward the Michigan synclinal basin throughout much of Paleozoic time.

Rocks of the Distributive Province.—Ohio and Indiana: The rocks of these two states which are available as source material for Michigan de-

posits lie on the western flank of the Ohio anticline, and on the eastern flank of the Indiana uplift. Available material is of sedimentary origin and made up principally of limestone.

Wisconsin: The deposits of southern and central Wisconsin are Paleozoic. They form a great semicircle around the pre-Cambrian peneplain of the central and northern parts of the state. The Cambrian of Wisconsin is a sandstone, and the Ordovician and Silurian strata are limestones.

Lake Superior Region: The rocks are crystallines of both igneous and sedimentary origin, and Keweenawan clastics, volcanics and plutonics.

Zircons, garnets, amphiboles, epidote, tourmaline, titanium minerals, and occasionally hypersthene and enstatite have been found in the Lake Superior crystalline rocks. They are usually recorded as occurring in gneisses, schists, and metamorphosed graywacke.

Pre-Cambrian of Canada: The crystalline rocks in the region adjoining eastern Michigan are granite-gneisses, gneisses, granite, and some schists. The younger Huronian rocks consist of clastics and eruptives.

The minerals recorded from this district of Canada are hornblende, epidote, biotite, chlorite, zircon, garnet, ilmenite accompanied by leucoxene, rutile, and tourmaline. Of these species zircon, epidote, chlorite and hornblende are most frequent. Garnets are rare, occurring only in garnetiferous schists.

Ontario Peninsula: Malcolm (1915, p. 5, 9) describes the rocks of Ontario, Canada. Limestones, dolomites, and shales of the Ordovician, Silurian, and Devonian periods rest upon formations of pre-Cambrian age. A few feet of sandstone and arkose, possibly Potsdam, occurs at the base of the section.

Summary of Rock Types.—From the southerly direction only limestones and shales are available source material for Marshall sediments. Possibilities range from middle and upper Ordovician through the Devonian, with emphasis on the Devonian, since that formation seems to have been the most widespread.

From the west and northwest, in addition to the Ordovician and Silurian limestones, Cambrian and Ordovician sandstones are possible sources. In the northwest, north, and northeast crystalline rocks are also exposed and were undoubtedly the original rocks from which came most of the Marshall sandstone. To the east were Ordovician, Silurian, and Devonian formations, and crystalline rocks as source materials.

Source of Marshall Sediments.—Mechanical sediments derived from the south are very rare, at least within the triangular area adjacent to Michigan, and bounded by the Cincinnati arch. The thin layers of the Sylvania and Pendleton sandstones of Ohio and Indiana respectively, are the only sources of sandstone. These formations could not, of course, have contributed any appreciable amount of sand toward the thick Marshall section.

Drainage off the Cincinnati anticline into the Michigan basin must have brought in large amounts of chemical sediments, and possibly some silt. Any other material would be out of the question due to a lack of source material in the distributive province.

From the standpoint of available material the southern sources prove decidedly inadequate.

Condit (1912, pp. 153-163) has studied Ohio sands petrographically and from his microscopic work there is further evidence that Ohio sandstones are not closely related to the Marshall formation.

The sandstones of Ohio from the Permo-Carboniferous and older formations contain minerals such as quartz, muscovite, kaolin, microcline, zircon, limonite, orthoclase, plagioclase, sericite, hematite, apatite, chlorite, pyroxene (altered), pyrite and marcasite.

Condit (1912, p. 159) says that the scarcity of garnet is characteristic of all sandstones of, and older than, Carboniferous age which had been studied at that time.

On the western side of Michigan the Wisconsin and Lake Superior regions provide an adequate primary source for mechanical sediments in the pre-Cambrian crystalline rocks. The immediate source may have been the Cambrian sandstones and Ordovician limestones.

Two of the lower members of the Cambrian sandstone (the Jordan and Madison) of Wisconsin have been studied petrographically by Ockerman (1930, pp. 346-354). He finds (p. 350) that 90 per cent of the residues of the eastern Wisconsin sandstone consists of garnet, as compared to 75 to 80 per cent for the residue from the western sandstone. Zircon is next most abundant, with tourmaline third. This high percentage for garnet could hardly persist during a reworking of the sandstone, but in the western Marshall sandstone garnet still predominates over tourmaline and zircon.

In addition to the abundance of garnet, there is another possible indication of the important influence of metamorphic rocks in the presence of pink to purplish zircon in the western Marshall residues.

On the other hand, rarity of garnet and the absence of purple zircons in the eastern Marshall sediments, suggest that the most important source rock was igneous rather than metamorphic.

Mackie (1923, p. 201) observed purple zircon in specimens of gneiss from Canada, but found only colorless zircon in the granite-gneiss which resembles granite in every particular except that it is more or less foliated. Since the Laurentian granite-gneiss is the most widespread of the crystalline rocks of the Canadian region under consideration it probably was the chief source of the eastern Marshall sediments, whereas true gneisses and schists contributed the major portion of the western Marshall sediments. However, until samples of these crystalline rocks have been examined by crushing and study of the heavy residues no more definite conclusion can be reached.

The presence of hornblende, actinolite, and epidote in the the western residues indicates that their source was comparatively near at hand, because these ferromagnesian minerals are too unstable to endure prolonged transportation.

Scarcity of ferromagnesian minerals, the advanced stage of alteration of such of these minerals as do occur,

and the presence of leucoxene almost to the exclusion of ilmenite point to a complicated history of weathering, and prolonged transportation before the deposition of the sediments in the Marshall sea.

Either the north end of Lake Huron or the region of the Frontenac axis is far enough away from Michigan to account for a long period of transportation. Such a distant source makes it difficult to determine just when the major portion of the weathering took place, but although much of the decomposition must have been accomplished during the transportation, undoubtedly some preceded it.

Topographic irregularities at the top of the upper Marshall indicate that the younger part of the formation was exposed to agents of weathering and erosion after deposition, and some alteration to gibbsite has taken place.

Summary of Rock Sources of Marshall Sediments.—The Wisconsin and Upper Peninsula rocks have furnished vast quantities of sediment to the western and southern deposits of Marshall sandstone. The rocks of Canada, primarily the Laurentian granitic-gneiss and the débris therefrom, furnished the major portion of the material for the eastern deposits of Marshall sandstone.

The northern extension of Marshall is as yet almost untouched by the drilling program, and it can only be surmised that the source material resembles that of the Lake County well.

The Paleozoic rocks have not been able to contribute mechanical sediments in large enough quantities to be appreciable in the deposition of Marshall sediments, but these areas probably provided cementing material.

Climate.—Were the Marshall formation derived directly from crystalline rocks, the degree of alteration of the feldspars would be a means of postulating climatic conditions for the period. However, the source of the Marshall is somewhat indirect and much of the kaolinization must have occurred during previous erosion cycles.

Due to decomposition, feldspars are not easily identified in Marshall residues, and the small amount still fresh may show direct origin in the pre-Cambrian rocks.

Since hornblende is still fresh in the western wells it may suggest that the Marshall climate was unfavorable to decomposition. However, since amphiboles are so abundant in the source rock, it is more probable that hornblende indicates a direct source from the hornblendic schists of the Laurentian and Huronian rocks.

The alteration which caused the formation of gibbsite was undoubtedly accomplished in a warm climate. Evidence of high temperature and aridity toward the close of the Marshall are found in the saline, gypsum, and lime deposits which succeed the Marshall formation.

Transportation and Deposition.—The uniform extent and thickness of the Marshall formation indicates that a well developed drainage system must have been responsible for its accumulation.

Probably the residual soil of the distributive provinces was removed and deposited during Coldwater and lower Marshall times. As rock disintegration

became increasingly important during Marshall time due to gradual uplift, bed rock itself was eroded. Thus the upper Marshall includes both unweathered débris and some products of alteration. The western deposits of the Marshall sandstone contain both the red ferric hydroxide products of decomposition, and fresh ferromagnesian minerals.

The eastern deposits of Marshall are colored by iron minerals only in the older part of the formation, probably because iron sources were not so abundant to the east.

Heavy residues show no more relationship between the eastern and western sandstones of red color, than between those of buff and red colors, showing that the rock colors in themselves are an inadequate basis for supposing that the lower Marshall of the two sides of the state had the same origin.

Grain size determinations indicate fairly uniform textures within the 0.10 mm. and 0.50 mm. limits. The quartz grains are angular to subangular and suggest transportation by water.

After, and possibly during the deposition of the Marshall formation, precipitation of secondary quartz took place, and along with calcite filled the interstices between sand grains. Pyrite, some magnetite, and possibly marcasite were abundantly precipitated. The pyrite seems to have had a special affinity for the solution pits of quartz grains. In some of the sediments celestite was also deposited in small amounts.

CONCLUSIONS

From a study of the heavy residues of the Marshall sandstone it has been determined that:

1.—The formation was derived chiefly from the Laurentian and Huronian rocks and the débris therefrom (such as the Cambrian and Ordovician sandstones).

2.—The source of the western sediments was nearer than the source of the eastern sediments. This condition resulted in differences in the mineral assemblages of the two regions.

3.—The residues from southern Michigan sediments are more closely allied to the western than to the eastern residues, undoubtedly because of structural conditions. Livingston was hardly affected by possible southern sources due to the type of rock in the southern distributive province.

4.—The sandstone of Marshall time was a marine deposit, owing its origin chiefly to the material contributed by a well developed drainage system.

5.—The depositional basin was areally extensive, and gradually became more shallow throughout the period. Much of the land appeared above water during the end phase of upper Marshall deposition, and erosion caused topographic irregularities between the Marshall, and the succeeding Michigan formation deposits.

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HEAVY MINERALS OF THE PLEISTOCENE BEDS OF THE DON VALLEY, TORONTO, ONTARIO

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ABSTRACT

A study was made of the heavy minerals from the glacial and interglacial deposits of Pleistocene age exposed in the Don Valley, Toronto, Ontario. The mineral occurrences are listed and special characteristics noted. The actual mineral occurrences are very similar in glacial and interglacial beds but when the relative abundance of certain minerals is plotted as percentage of total heavy minerals present, curves are obtained showing a rather sharp difference between glacial and interglacial beds and a very close resemblance between the various interglacial beds examined. In all cases the mineral assemblage is of typically Pre-Cambrian derivation. It is thought that the materials of the interglacial beds were derived, either from the glaciers and icebergs themselves, or from older boulder clays, and very little from the Paleozoic sediments which underlie the region.

A succession of samples were collected in the Don Valley, mainly from the Don Valley Brickyard, with a view to examining their heavy minerals. Although the majority of the heavy minerals of the whole succession have been derived from igneous and metamorphic rocks of the pre-Cambrian shield, it was thought possible that changes in the direction of ice movement, or, in the inter-glacial periods, of the river drainage, might result in a different suite of heavy minerals being deposited.

TREATMENT OF MATERIAL

The treatment used was the standard one for heavy mineral separation. In the case of sands the sample was screened to remove the coarsest material, washed in water, and then treated with hydrochloric acid (up to 50 per cent) to remove carbonate, which is generally abundant in these beds, except in the coarsest sands. The residue was then treated in a separation funnel in Thoulet solution (Potassium-Mercuric Iodide), slightly diluted to give a specific gravity of 2.95. The heavy minerals were caught on a filter paper, washed in water and alcohol, dried, and mounted either on a temporary slide using different refractive index liquids as the mounting medium, or as a permanent slide using Canada balsam.

In the case of clays, the finest clayey material was washed out and the silty or sandy residue treated in the same way as the sands.

MINERAL CHARACTERISTICS OF THE BEDS

Table I shows the heavy mineral occurrences and their relative abundance in the different beds. The figures represent approximate percentages of the total heavy minerals present. Characteristic features of the beds are described below.

TABLE I, *showing the relative abundance of mineral occurrences expressed as percentages of total heavy minerals. One per cent on the table indicates one per cent or anything less than one.*

Specimen No.	Horizon		Apatite	Augite	Diopside	Dolomite	Enstatite	Epidote	Garnet	Hornblende	Hypersthene	Kyanite	Leucoxene	Magnetite & Ilmenite	Titanite	Topaz	Tourmaline	Zircon	Zoisite	Other rare occurrences
26	1st Boulder clay		6	1	2		2	1	18	42	6	1		11	2			1		Rutile Biotite
1	Lower Don sands	1st Interglacial		5	6	3	2	3	16	40	3			13	1	2		4		
2				5	5	3		1	29	18	8		1	26	1					Hematite
3				5	2		1		25	44	5	1	1	7	1	2		4		Barite
4			2	8	2	2	2		23	49	3	2		8	2		1	1		
5	Don clay		4	5	1		1		22	36	4	2		21	3		1	5		Chlorite
6	Upper Don sands			5	6	1	1	1	36	30	8	1	1	8	1	1		1	1	Monazite
8			3	6	10			3	21	35	2	1		12				2		
9			3	8	2		1	3	28	37	5		1	6	1			2	2	Staurolite Hematite Sulphides
24	Scarboro clay			4	6	4	1	1	28	42	4	1		9				3		Actinolite
21				1		1	1	5	21	33	3	2		27				7		Corundum?
22	2nd Boulder clay		1	2	3			2	15	55	6	1		10	1	1		4		
23	Varved clay	2nd Interglacial	3	4	3			1	30	33	8			13	3			2		
25			2	3	2	2	1	4	26	37	3	1	2	11	3			4		
27			1	11	11		3	1	26	33	6			5	2			2		Staurolite Andalusite
20			2	3	2		2	2	23	28	1	1	1	28	3			5	1	

First Boulder Clay (26).—This clay lies immediately above the Dundas and contains abundant fragments of the shale and impure limestone of that formation. The heavy mineral grains are angular and coarse, apparently all derived directly from the pre-Cambrian and not from reworked Paleozoic sediments. Material from the latter is, of course, shown up in the abundant carbonate dissolved in the early part of the laboratory treatment. In mineral content the high hornblende and relatively low garnet percentage are the chief features.

Don Beds (1).—Lower sand (1-4). This sand varies very much in texture, some beds being very coarse, almost gravel. Interbedded with the sand are narrow bands of pinkish clay. The heavy mineral percentages vary considerably in these different beds, but all grains show better rounding than those of the underlying boulder clay. In general the coarse sand has a high garnet-magnetite-ilmenite percentage and relatively low hornblende, while the finer sands are characterized by high hornblende content.

(2) Clay (5). A clay bed about 5 feet thick occurs above the sands just mentioned. Apart from the rather smaller size of the grains and the high magnetite-ilmenite percentage, the heavy mineral content differs very little from that of the sands above and below.

(3) Upper Sands (6, 8). Some

coarse, reddish sand occurs above the clay just mentioned and below the Scarborough clay. The heavy mineral grains are large and moderately well-rounded, but show no very distinct features by which the beds might be recognized.

Scarborough Clay (9, 24, 21).—The heavy minerals of this clay, although of smaller size, differ little in appearance and relative abundance from the Don beds. One exception is the presence of some very well-rounded zircon grains which have been worn so much more than the associated minerals that it seems probable that they were derived by the reworking of some Paleozoic sediment, rather than directly from the pre-Cambrian rocks. The greater part, however, of the grains are evidently derived from the pre-Cambrian and are not much worn.

Second Boulder Clay (22).—This bed varies in the Don Valley from the thickness of a few boulders up to 5 or 6 feet. The heavy mineral grains vary very much in size and degree of rounding. The hornblende content is extremely high (55 per cent) while the garnet content is correspondingly low. Most of the minerals are angular, but a few small, round zircon grains occur. The outstanding feature is the number and variety in size of the hornblende grains.

Varved Clay (Second Interglacial) (23, 25, 27, 20). The varved clay, although made up mainly of very fine material, contains a fair proportion of relatively coarse heavy minerals. It is probable that a good proportion of this was dropped from floating ice broken off from the glaciers, which may explain the rather close similarity of the heavy mineral assemblage with that of the boulder clays. The hornblende content is fairly constant at about 33 per cent and garnet at rather less. Magnetite and ilmenite are less abundant than in the lower beds, and pyroxenes are relatively common. The higher part of the 80 feet of varved clay contains fewer coarse grains and a higher percentage of magnetite and ilmenite, but otherwise differs little from the lower part overlying the second boulder clay.

FEATURES OF THE MINERAL SPECIES OBSERVED

Actinolite. Rare, in long, pale-green prisms. Some actinolite may be included in the hornblende count.

Andalusite. Very rare. Only identified in one specimen from the varved clay.

Apatite. Occurs generally in clear, colorless, well-rounded grains, sometimes with inclusions. It is a constant, but not common, mineral in these sediments.

Augite. A fairly common mineral in some slides. It occurs in oval, pale-green grains derived from stumpy prismatic crystals. The deep-green variety is rare.

Barite. Very rare. One grain was observed in the Don sands.

Biotite. A rare mineral, though its apparent rarity may be partly explained by the fact that it does not sink easily in the solution used.

Chlorite. Only a few flakes were observed.

Diopside. A fairly common mineral which cannot be sharply differentiated

from pale augite. It occurs in the same oval to stumpy prismatic grains as the latter, is colorless to very pale-green, and shows good cleavage and a high extinction angle.

Dolomite. Some dusky carbonate grains not dissolved in the acid digest may be dolomite.

Enstatite. In colorless or very faint pleochroic green to pink grains, showing good cleavage and parallel extinction. It grades into the pale variety of hypersthene.

Epidote. In irregular, pale- to light-green, or sometimes brown, grains. It shows bright-red and green interference colors and usually a "compass needle" effect in the interference figure.

Garnet. The second most common heavy mineral in these sediments. The grains vary a great deal in color from deep-pink or brown to colorless, and in shape from angular fragments and grains showing rectangular "step fracture" to those which are moderately well-rounded. It is possible that some colorless spinel has been listed as colorless garnet, as these minerals are very hard to distinguish when mounted in balsam.

Hornblende. The commonest of the heavy minerals in this series. It occurs in dark-green, pleochroic grains, which may be rounded stout prisms or angular splinters. Under the term "hornblende" are included all the green monoclinic amphiboles, except actinolite which has been differentiated in one or two samples.

Hypersthene. Unexpectedly common. This feature is characteristic of Canadian sediments in general, as noted by Fraser, due probably to the prevalence of hypersthene in the widespread pre-Cambrian gneisses, a prevalence not fully recognized in thin section work. The hypersthene occurs usually in somewhat rounded prismatic grains, but sometimes also in angular chips. Pleochroism is often beautifully marked, from deep red to grass green.

Hematite. In irregular grains, but not common.

Kyanite. This mineral is easily recognized by its stout prismatic habit with cleavage at right angles to the prism length, and oblique extinction. It is not very common in any one bed but is present in most of them. The grains are not much worn.

Leucoxene. Not a common mineral in these beds. The ilmenite, which is fairly common, is evidently not much altered to leucoxene.

Magnetite and Ilmenite. These two minerals are classed together in counting because, although easy to separate with the aid of a magnet, they are hard to distinguish when mounted in balsam on a slide, where the count is made. It is probable that at least half the combined percentage listed is magnetite. Both minerals are somewhat rounded but very little oxidized.

Monazite. This mineral is only a rare occurrence and cannot be said to be typical of these beds.

Rutile. Rutile is surprisingly rare, considering its wide distribution among the heavy minerals of most sediments. The grains observed were the deep, foxey red type, and rather angular.

Staurolite. Occurs in yellow, pleochroic grains, showing low birefringence, but is not very common. It was

seen in the lower part of the Scarborough clay and in the upper part of the varved clay.

Titanite. Titanite is not easy to identify owing to the number of different types of occurrence. It may occur in clear, colorless crystals in all degrees of rounding, or in irregular or rounded grains of a fairly deep brown color. It seems to be a fairly steady occurrence, forming up to 3 per cent of the total heavy minerals. The very high birefringence and high refractive index are its strongest characteristics.

Topaz. This mineral is not common, but is sometimes seen in rather angular, colorless grains.

Tourmaline. Tourmaline is much less common here than in most sediments. In most sediments it would probably come among the five most common heavy minerals, but in these beds it is an exception to find it at all. It is interesting to note how common it is in the Dundas hard bands of Toronto, the minerals of which were apparently derived from the same general locality, but at a much earlier stage of erosion when more pneumatolytic minerals were exposed at the surface.

Zircon. This mineral too is rarer than usual, although there are nearly always a few grains in any specimen. Most of the grains observed were clear fragments of good crystals, often with bubbles or inclusions. These show comparatively little rounding. Another type, much more worn, and often dusky and zoned, may have been derived by the reworking of some older sediments, since they seem to have undergone much more abrasion than the other mineral grains.

Zoisite. Not very common, but seen sometimes in rather rounded grains which may show the typical deep blue anomalous interference colors. Like epidote, these grains generally show a "compass needle" interference figure.

CONCLUSIONS

To a certain extent the results of this investigation are disappointing in that there are no special minerals or physical features of minerals which are diagnostic of particular beds. Much the same mineral assemblage, evidently derived from the same general source, the pre-Cambrian rocks to the north and northeast, is seen in all the beds examined. No doubt some of the interglacial material results from the reworking of earlier glacial or interglacial deposits, but the transportation in the second journey has not been long enough to cause rounding which might make them conspicuous from the material derived directly from the pre-Cambrian shield.

We have, therefore, to fall back on the relative abundance of individual minerals to find criteria for differentiating one bed from another. This relative abundance is obtained by identifying and counting between 100 and 200 grains on a slide and expressing the result as percentages of total heavy minerals. Using this criterion, although there are differences between different horizons of the same member, an average of the samples of each member (Don, Scarborough, etc.), shows a very close resemblance between the interglacial beds as opposed to the boulder clays.

These differences are best shown up

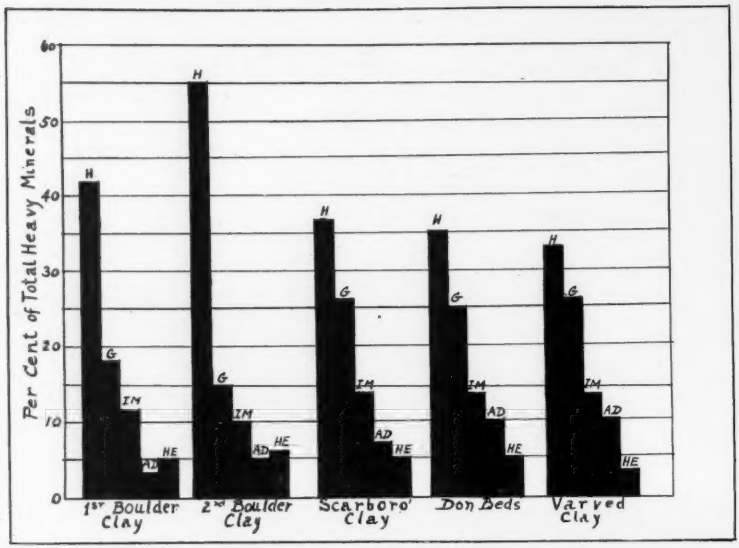

FIG. 1. Chart showing abundance of heavy minerals for each stratigraphic member expressed as percentages of total heavy minerals. H—hornblende, G—garnet, IM—ilmenite and magnetite, AD—augite and diopside, HE—hypersthene and enstatite.

on a graph (fig. 1) where the percentage occurrences of hornblende, the combined monoclinic pyroxenes (augite and diopside), the combined orthorhombic pyroxenes (hypersthene and enstatite), garnet, and combined magnetite and ilmenite, are plotted for each stratigraphic member. The values have been obtained from the averages of several samples of each member. It may be seen that the curves for the boulder clays show higher hornblende percentages and lower garnet and magnetite than do the interglacial beds. The close similarity between the three interglacial members is very striking.

From the similarity of the actual mineral species in the glacial and interglacial beds, it is suggested that most of the material deposited in the interglacial lakes was derived either directly from the glaciers or icebergs, or from the reworking of the previous boulder clay, and that very little material was provided by the underlying rocks which the interglacial drainage might have eroded.

The writer wishes to thank Prof. A. MacLean of the University of Toronto, for criticism and advice. He is also indebted to Messers A. Holstein and E. W. Shaw for assistance in collecting material.

JOURNAL OF SEDIMENTARY PETROLOGY, VOL. 3, NO. 3, PP. 119-120, FIG. 1
DECEMBER, 1933

THE PREPARATION OF CLAY SAMPLES FOR ELUTRIATION BY STEAM AGITATION

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ABSTRACT

The article describes a method of preparing clay samples for elutriation. The samples are agitated by means of a jet of live steam under pressure and ten minutes usually suffices for normal clays.

The writer has been engaged for some time in the mechanical analysis of samples taken from the recent deposits of New Mexico, and was troubled considerably when dealing with clays, in that flocculated clay material in the form of balls contaminated the pure sand grains as separated by the elutriator. Agitating and boiling with the usual deflocculating agents did not entirely eliminate the trouble. The method which was finally used, and which gave by far the most satisfactory results was suggested by Mr. G. I. Bathe of Philadelphia. By this method clay samples may be quickly prepared for elutriation by agitating them in a flask by means of a jet of live steam under pressure. The procedure and apparatus necessary to accomplish this is simple.

The apparatus consists of a small boiler, 8 inches long by 2 inches in diameter with a capacity of 250 cubic centimeters, capable of delivering a continuous jet of steam for 10 minutes at about 12 pounds pressure; it is equipped with a safety valve, a pressure gauge, and a shut-off valve. Steam is raised by an ordinary Bunsen burner. The flask, which may be of any convenient size, used in conjunction with the boiler is fitted with a two hole rubber stopper to accommodate the inlet pipe from the boiler and the exhaust tube. The inlet tube and the jet are made of copper pipe having an outside diameter of one-eighth of an inch; the exhaust tube is glass, and of a slightly larger diameter than the inlet tube. The jet is set at the bottom of the flask so as to give a circular swirling motion to the sample. The set up may readily be seen from the illustration.

To prepare for agitation steam is raised in the boiler till the pressure gauge reads about 30 pounds, at which point the safety valve may be set to blow off. About 20 grams of the clay sample are put into the flask, the stopper is inserted, and the steam is turned on. An equal quantity of sodium carbonate may be added to assist de-flocculation. Condensed steam soon adds sufficient water to allow the sample to flow smoothly around the flask under the swirling motion imparted to it by the steam jet. The steam jet may be left open for the full 10 minutes of

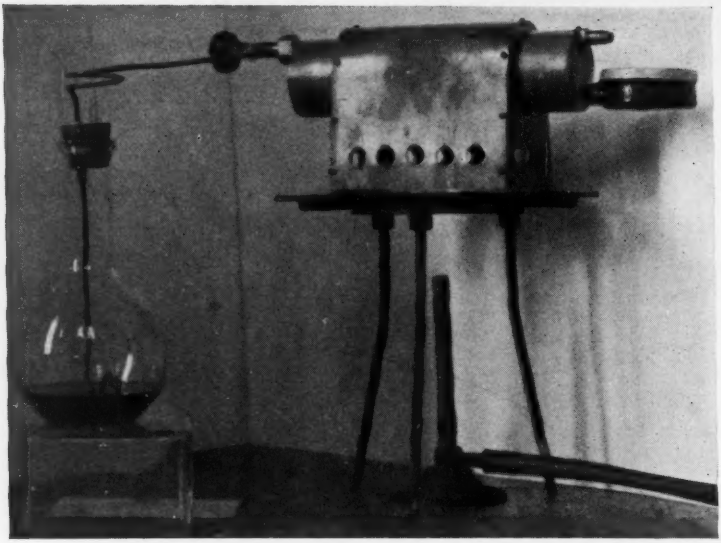

FIG. 1. Apparatus for preparation of clay samples for elutriation.

agitation, or the shut-off valve may be closed every two minutes or so to permit the pressure to build up to 30 pounds again, thus giving discontinuous bursts of agitation at higher pressures than are obtained by allowing a continuous flow of steam.

One "steaming" for ten minutes is usually sufficient to break up the clay material in normal clays so that it will wash out through the overflow tube of a Crook's elutriator, leaving the clean mineral grains uncontaminated by "clay balls." However, very fine clays often prove more refractory and may have to be carefully drained from the elutriator and reagitated in the steam flask in order to completely rid the mineral matter of clay material. A very fine clay from New Mexico was returned to the steam flask four times before the sand grains were entirely uncontaminated by flocculated clay material. This trouble might be eliminated by using a larger boiler with a correspondingly longer steaming capacity.

One caution must be noted when quantitative work is being done; after steaming a small amount of the sample will be found adhering to the jet pipe inside the flask. This may be collected by holding an evaporating dish beneath the jet, after the flask has been removed, and washing it carefully with a wash bottle. The washings may then be added to the main charge of the elutriator.

JOURNAL OF SEDIMENTARY PETROLOGY, VOL. 3, NO. 3, PP. 121-135, FIGS. 1-5,
DECEMBER, 1933

THE DISPERSION OF FINE-GRAINED SEDIMENTS FOR MECHANICAL ANALYSIS

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ABSTRACT

The purpose of dispersion is to destroy all aggregates without affecting the size of the individual particles. Dispersion procedures used or recommended for soils or sediments are reviewed in terms of their contributions to this end. The general subject of dispersion is then related to the underlying principles, and a group of procedures which most nearly accord with these principles are experimentally compared. From the results obtained, a general routine of dispersion for fine-grained sediments is offered. The routine is arranged in two sequences, and includes soaking in water, rubbing with a brush, shaking, heating to boiling, and washing out foreign electrolytes. The particular sequence used, and the steps involved, depend on the nature of the sediment.

The dispersion of sediments into their individual particles is prerequisite to any method of mechanical analysis. Among coarse sediments the preliminary treatment may involve only drying them in preparation for sieving, but if the sediment is indurated or consists mainly of fine grains, dispersion becomes more difficult.

The present paper is concerned with dispersion procedures for fine-grained clastic sediments in which the bulk of the material is below about 0.06 mm. in diameter. Clay, shale, silt, siltstone, and sediments like till which have a range of sizes from coarse to fine, are included in this category. In an earlier paper the writer (11)[1] suggested a dispersion technique for unconsolidated sediments, particularly till and clay. The routine described in the present paper is a modified generalization of the other, designed to extend its usefulness to a wider range of sedimentary types.

The purpose of dispersion is to separate all aggregates into individual particles[2] without affecting the size of the individual particles themselves. Thus the dispersive treatment must be sufficiently vigorous to destroy aggregates, but gentle enough not to break crystal fragments. The development of a method which lies between these limits is obviously difficult, and may never be achieved in actual practice.

Soil scientists have devoted considerable attention to dispersion, and have developed numerous techniques for the purpose. A review of these techniques will indicate the range of treatments

[1] The numbers in parentheses refer to the bibliography arranged at the end of this paper.

[2] The contention has been raised in the past that thorough dispersion is not always advisable, on the ground that the original sediment may have been partially or completely flocculated. It is quite generally agreed at present that mechanical analysis based on complete dispersion is of more significance since it furnishes basic information on the ultimate distribution of particle sizes in the sediment.

available, and from them may be chosen the procedures most applicable to sediments. Some of the techniques are entirely physical or chemical, but combinations are most common. Many techniques include several steps or procedures, but for review purposes it is more convenient to consider each procedure individually.

DISPERSION PROCEDURES

A. PHYSICAL

Prolonged Soaking in Water.—The sample is crushed into small lumps and allowed to soak either in water or in dilute solutions of electrolytes.[8] The period of soaking allows each particle to be surrounded by a film of water, or fills the pores of the rock, and so loosens the grains and aids in their dispersion. The method is particularly suitable for partially indurated sediments, and was used by Rubey (19) in his study of Cretaceous shales. He reviewed previous work and emphasized that the ease of disintegration varies with the moisture content of the sample. Rubey soaked his samples in dilute ammonia for 8 weeks, but pointed out that prolonged soaking may dissolve fine particles and hydrate minerals. In general, the period of soaking depends on the degree of consolidation of the sediment. Dragan (6) recommended a 24-hour period of soaking in distilled water as advantageous to the dispersion of soils.

Rubbing or Trituration in Water.—The sample is made into a paste with water and rubbed with the finger, with a stiff brush, or triturated with a rubber pestle. Water is added from time to time, and the dispersed material poured into a beaker, until all the aggregates are destroyed. Whittles (22) emphasized the necessity of wetting the samples gradually so that the water penetrates them throughout. The general method has found considerable favor among soil scientists.

Shaking in Water.—Shaking in water or in dilute electrolytes is a widely used dispersion procedure. Reciprocating, end-over-end, and rotary shakers are commonly used. Joseph and Snow (10) considered reciprocating shakers preferable. The periods of shaking vary from 1 to 24 hours. Richter (18) observed some breaking of grains during shaking, and Nolte (13) compared the size reduction effects of shaking and boiling on carefully separated grades. He found less reduction from shaking than from boiling, although he warned against too long a period of shaking. Olmstead, Alexander, and Middleton (16) considered the breaking of particles to be negligible even after 16 hours of shaking. Hissink (8) contended that rubbing with a brush had as great a grinding effect as shaking.

A detailed study of shaking was made by Puri and Keen (17) in 1925. They shook soil samples in water for varying lengths of time and measured the degree of dispersion by the percentage of fine material set free. This percentage, called the "dispersion factor," increased rapidly at first, and then more slowly. The curve was

[8] While the use of electrolytes is a chemical procedure, it is mentioned here as a variation which often accompanies this and following physical procedures. In the discussion, however, the effects of the electrolyte are not considered, since that subject will be treated under a separate head.

found to agree with the equation $d = a + k \log t$, where d is the dispersion factor, a and k are constants, and t is the time. The test periods extended over intervals as great as 100 hours, but in no case did the dispersion factor reach an upper limit. Dispersion thus seems to be a continuous function of the time, but a 24-hour period of shaking was found sufficient to carry the degree of dispersion over the steep part of the curve. The original moisture content of the sample and the concentration of the suspension were found materially to affect dispersion.

Experiments by the writer showed that the effectiveness of shaking also depends on the presence of coarse material in the sediment. A lake clay, having no particles above 0.03 mm. diameter, contained undispersed clay pellets after 6 hours of shaking, while a till with considerable sand became fully disaggregated within an hour. Davis (4) used rubber balls to hasten the dispersion of fine material, and glass beads have also been used. The effect is similar to the sand in till, and reduces the time of shaking to a fraction of its previous length. Comparative analyses of two samples of the lake clay, one of which was shaken for 12 hours without glass beads, and the other 1 hour with them, showed no differences in the size distribution beyond the limits of experimental error, so that the grinding effect of the beads appears to be negligible.

Stirring in Water.—In 1927 Bouyoucos (1) used an electric drink mixer for dispersing soils. Due to the high speed of the stirrer, wire baffles were placed in the cup to prevent circular motion of the suspension. More recently Bouyoucos (2) compared stirring with shaking and found that 10 minutes of the former was more effective than 16 hours of the latter. In sandy soils he noticed some apparent breaking of the sand grains from more prolonged stirring. The stirrer has thus been found to be one of the most effective dispersion devices, and Joseph and Snow (10) considered it as having excellent possibilities for the purpose.

Vibration in Water.—The dispersion of samples by vibration is one of the newer procedures. In 1924 Whittles (22) developed a mechanism in which a rapidly vibrating hammer struck the celluloid bottom of a glass cylinder at controlled frequencies and amplitudes. A fair degree of dispersion was obtained from trituration with a rubber pestle, followed by vibration for 1 hour at 10,000 vibrations per minute.

In 1931 Olmstead (15) used supersonic waves for dispersion. The vibrations are produced by a piezoelectric quartz crystal immersed in an oil bath, and energized by a vacuum tube oscillator. The energy is transmitted through the oil into a flask containing the soil suspension. The sample is vibrated for several 2-minute periods, followed by decantation of the dispersed material. Olmstead compared his method with rubbing and found a close agreement, but the supersonic treatment was much more rapid.

Ignition.—Heating samples in the dry state has been used for disaggregation by some workers. Nolte (13) mentioned the far-reaching physical and chemical effects of such treatment, and

Richter (18) found that among several procedures, ignition gave the worst results due to the destruction of colloids and the possible fusion of grains.

Boiling in Water.—The boiling of samples in water or in dilute electrolytes is a procedure about which more controversy has taken place than about any other process of dispersion. In 1919 Nolte (13) reviewed the history of the subject, and furnished a summary of the situation up to that year. He finally concluded that boiling could not be recommended because it simultaneously reduced the size of the larger particles and flocculated the smaller ones. In the same year Odén (14) observed that boiling destroyed aggregates larger than 10 microns, while particles smaller than 1 micron flocculated to aggregates between 1 and 2 microns in size. In 1927 Von Hahn (9) referred to boiling as a "barbaric practice" because of its physical and chemical effects on the suspension.

The most important contribution was made by Wiegner (23) in 1927. He compared the effects of boiling on samples in which the water-soluble salts were either washed out or left in. He found that in the washed samples the dispersion was increased by boiling, while the electrolytes in the unwashed soil caused flocculation. Thus he showed that the same treatment may either prevent or aid dispersion, depending on the presence or absence of appreciable amounts of foreign electrolytes.

Periods of boiling have varied from about 10 minutes to more than 40 hours. It is doubtful whether boiling for 24 hours or more aids the dispersion, even if no foreign electrolytes are present. Wiegner considered an hour to be sufficient; the writer (11) heats the suspensions to the boiling point, but does not allow them to boil. In this manner the agitation due to heating is able to perform its function of dispersion without the disadvantages that may follow a prolonged application of heat.

Removal of Water-Soluble Salts.—The washing out of foreign electrolytes, as a preliminary treatment of samples, has received increasing attention in recent years. The process may be accomplished with an unglazed porcelain filter of the Pasteur-Chamberland type. The sample is soaked in distilled water, with occasional stirring, and the liquid filtered through the porcelain by suction. The process is repeated if necessary. By removing the water-soluble salts, the material is more readily dispersed, as Wiegner proved in the case of boiling. According to Olmstead, Alexander, and Middleton (16), electrodialysis may also be used for dispersion.

B. CHEMICAL PROCEDURES

Leaching in Acids or Alkalies.—Leaching in dilute acids as a preliminary treatment has long been used by soil scientists, but questions of its advisability in all cases have recently been raised. From the viewpoint of sedimentary petrology it cannot be emphasized too strongly that all primary carbonate particles should be retained during mechanical analysis because they are an integral part of the size frequency distribution. When only secondary carbonates are present they

may be removed, but when both primary and secondary carbonates are involved, the one cannot be removed without destroying the other, and the decision to remove all or none must depend on the problem in hand.

The digestion of samples in alkalies, such as sodium hydroxide, has also been practised. Such treatment is in the same category as acid treatment from the viewpoint of sedimentary petrology.

Peptization with Very Dilute Electrolytes.—The use of small amounts of peptizing electrolytes, such as sodium or ammonium hydroxide, sodium carbonate, or sodium oxalate, is very widespread, and few techniques do not include one or another of them. The amount of peptizer used varies widely among workers. Sodium carbonate, for example, has been used in concentrations from about 0.025 to 0.2 per cent. This wide range of usage suggests that there may be no single optimum concentration for all soils or sediments. In order to discuss this point, certain aspects of colloids are briefly reviewed here.

Colloidal suspensions differ from true solutions in that the latter are permanently stable, while the former are not necessarily so. It is generally accepted that in a stable colloidal suspension each particle has an electric charge arranged about it in a double ionic layer. The charge may be positive or negative, depending in part on the nature of the colloid. A detailed discussion is beyond the scope of this paper, and may be found in any reference work on colloids. Kruyt (12) discusses the subject at some length.

Under the influence of Brownian movement the particles are brought into the vicinity of others, but as long as the charges are above a critical potential the particles repulse each other, and adherence is prevented. If the charges are below the critical potential, or zero, the particles may adhere when they collide, with the result that aggregates are formed. These aggregates begin to settle and eventually the entire dispersed phase settles out of suspension as a flocculant precipitate. The rate of flocculation may be either slow or rapid, depending on whether or not any charges are present on the particles.

The magnitude of the charge varies appreciably with slight changes in the electrolyte content of the suspension, and the effect of a given electrolyte appears to vary with the nature of the colloid. In clays, for example, the charges may be reduced by adding calcium chloride, which removes or neutralizes the charges. Such electrolytes are called flocculants. Other electrolytes, as sodium carbonate, increase the charges on the clay particles, and are called peptizers. Beyond certain limits of concentration the peptizing electrolytes also cause flocculation, so that part of the distinction between the two types may be due to the relative concentrations necessary to produce flocculation.

Puri and Keen (17) studied the effects of several electrolytes on soil suspensions which had previously been washed free of soluble salts. Varying amounts of the electrolyte were added and the degree of dispersion measured and plotted. It was found that the sodi-

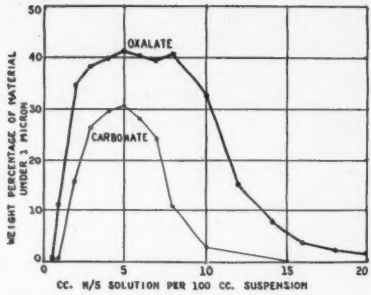


FIG. 1. Comparison of dispersive effects of sodium oxalate and sodium carbonate on lake clay.

um carbonate curve displayed a prominent plateau, which indicated that quite a range of concentration caused approximately the same degree of dispersion. This plateau-effect is a distinguishing feature of good peptizers because it allows some flexibility in the concentration that may be used. Other electrolytes, as KCl, had a much smaller dispersive effect and displayed a sharp peak at the optimum concentration. Puri and Keen concluded that the effects of electrolytes on suspensions are not abrupt, but cause a continuous change in the degree of dispersion as the electrolyte concentration is varied. They related their results to the phenomenon of base exchange, but no detailed explanation was attempted.

Winters and Harland (24) also studied the effects of sodium carbonate on dispersion. Their results agreed with Puri and Keen, and showed that the dispersive effects vary somewhat with the soil horizon.

Olmstead, Alexander, and Middleton (16) compared several peptizers, and decided that sodium oxalate was the most satisfactory. They pointed out that sodium and ammonium hydroxides yield good results when the calcium and magnesium carbonates have been removed by acid treatment and thorough washing. If calcium carbonate is present, sodium carbonate is better than either hydroxide because the carbonate decreases the solubility of the calcium carbonate, while the hydroxides produce flocculating calcium ions. Sodium oxalate, they found, was even better than sodium carbonate, because the calcium ions are completely removed by the oxalate. A comparison of the four peptizers on four soils showed that the oxalate had the greatest dispersive effect in every case.

The writer performed a series of experiments with sodium oxalate to determine whether it had a plateau-effect like sodium carbonate. Water suspensions containing 2.5 per cent of an unconsolidated calcareous Pleistocene lake clay were prepared by brush

TABLE I. *Dispersive effects of sodium oxalate and sodium carbonate*

Cc. of N/5 Electrolyte per 100 cc. Suspension	Amount of material under 1 Micron	
	Sodium Oxalate	Sodium Carbonate
0.5	1.6	trace
1.0	11.2	0.8
2	34.1	16.0
3	38.2	26.3
4	39.9	29.8
5	41.2	30.6
6	40.0	28.1
7	39.2	24.8
8	40.7	11.2
10	32.8	3.2
12	15.2	
14	8.0	
15		0.0
16	4.0	
18	2.4	
20	1.6	0.0

rubbing, and varying amounts of N/5 sodium oxalate or N/5 sodium carbonate were added. The per cent of material under 1 micron in the suspension was then measured by pipetting. Table I lists the data, and figure 1 shows the resulting curves. It is clear that sodium oxalate has both a greater dispersive effect and a wider range of safety than the carbonate. In both cases a concentration of about N/100 is optimum, as deduced from these curves.

From their study, Puri and Keen listed a concentration of N/25 sodium carbonate as optimum for soils. In an earlier series of tests by the writer on glacial till, a concentration of N/50 sodium carbonate was found to be optimum. Thus for sodium carbonate the optimum concentration varies from N/25 to N/100, depending on the material dispersed. The same relation appears in the case of the oxalate; the present study showed N/100 as optimum, while Olmstead, Alexander, and Middleton (16) recommended N/200 for soils. It thus appears that no single concentration is optimum in all cases, but in the writer's subsequent experiments a concentration of N/100 sodium oxalate was adopted, and yielded results that could be duplicated. In one or two instances, especially among highly weathered tills, the carbonate produced better dispersion than the oxalate, but for most other sediments the oxalate appears to be preferable.

The influence of base exchange phenomena on dispersion was investigated by Thomas (20). He found that soils in which the exchangeable bases were replaced by sodium were most readily dispersed by sodium carbonate, while a magnesium soil flocculated immediately on the addition of sodium carbonate, due probably to the release of magnesium ions. Because of the difficulty of preparing sodium-soils for routine purposes, Thomas recommended freeing the sample of exchangeable bases by acid treatment, followed by dispersion with sodium carbonate. From the viewpoint of sedimentary petrology, Thomas' work shows that since the exchangeable bases are at least partially replaced during dispersion, the presence of magnesium or other flocculating ions may hinder or prevent dispersion. The subject is quite complex, and requires research on the base exchange phenomena of sediments before the best treatment can be developed to offset these difficulties.

The general problem is further complicated by the fact that all sizes of particles are not equally sensitive to dispersion or flocculation. Tuorila (21) showed that in quartz suspensions the effects of flocculation begin to manifest themselves at diameters of about 0.04 mm., and become very pronounced between 5 and 10 microns, while particles under 4 microns are completely removed from suspension. Thus sediments made up predominantly of particles in the most sensitive range may be expected to be strongly affected by slight changes in the dispersion technique.

Most soils and sediments contain water-soluble salts in varying amounts, and Wiegner (23) showed that these foreign electrolytes have a marked effect on dispersion. He pointed out that if very small amounts are present, the

charges on the particles are above the critical potential, whereas if appreciable amounts are present, the charges are below the critical potential, and dispersion may be seriously hindered. Dispersion procedures, as shaking or boiling, increase the agitation of the particles and thus increase the number of collisions among them. Now if the charges are above the critical potential this added movement increases dispersion, while if they are below the critical potential the added collisions increase the rate of flocculation, and thus slow down or prevent dispersion.

Wiegner compared the effects on soils of (1) shaking for 6 hours, (2) rubbing for an hour with a brush, and (3) boiling for an hour with a reflux condenser. The analyses were conducted in a Wiegner tube, in which the same sample could be used after various treatments, because none of it is removed during the analysis. It was found that boiling was more effective than rubbing or shaking on washed soils, while shaking was the most effective on unwashed soils. The effect of N/10 ammonia was tested on washed and unwashed soils, and it was found that the washing out of the foreign electrolytes was more effective than the use of the peptizer on unwashed soils.

Wiegner's work shows that the safest general procedure to be followed is to wash the foreign electrolytes from the samples before dispersion. This is a rather tedious process for routine analyses, and apparently may be omitted except in cases where quite appreciable amounts of foreign salts are present. Tests by the writer on a number of sediments indicate that in the average case studied the water-soluble salts make up less than 0.1 per cent of the dry sediment.[4] In a suspension of 20 grams to a liter, this amounts to a concentration of about 0.002 per cent. Cations commonly present are Ca^{++}, Na^{+}, Fe^{+++}, and Mg^{++}. The common anions are SO_4^{--}, CO_3^{--}, and Cl^{-}. Gypsum appears to be the principal salt in non-calcareous sediments, while calcium carbonate or bicarbonate is more common in the calcareous types. These data indicate that for routine analyses many sediments do not require washing. In some cases, however, washing is clearly indicated, as with a black Pennsylvanian shale to be mentioned below, in which an appreciable amount of ferric sulphate was present. Unwashed samples flocculated on heating, while washed samples offered no difficulties.

GENERAL CRITIQUE OF DISPERSION

The foregoing discussion of the use of electrolytes in dispersion shows that the effect of a given electrolyte varies with its concentration, and that this effect is complicated by base exchange phenomena, by the sizes and composition of the particles, and by the presence of foreign electrolytes in the samples. In particular cases one or another of these factors may predominate, so that quite clearly no single technique can be advanced as equally suitable for all sediments or soils. Among the physical methods also there

[4] Soils from arid regions have a relatively large amount of soluble salts and it is possible that sediments may be similarly affected. The writer's samples are from a humid region.

is diversity of opinion concerning their relative effectiveness. Wiegner's work has shown that some of this diversity is due to the electrolyte content of the samples, but even with that allowance, there are still valid grounds for opinion concerning, say, the merits of shaking as against rubbing with a brush. The fact that there can be no single rigid dispersion method for all types of material has been stated in the past, and Davis and Middleton (5) particularly emphasized it.

There is another difficulty besides these. Dispersion has been found to be a continuous process, rapid at first, and slower later on. Puri and Keen showed this in their experiments on shaking, and Olmstead noted the same effect in his work on vibration. If this is universally true, it appears that complete dispersion can never be effected, or that there is a continuous increase of fine material due to the disruption or attrition of individual grains. Clark (3) recently published a paper on the problem, and he raised the question whether there exists in soils any unique size frequency distribution, or whether the distribution is not a function of the dispersion process. The difficulty of settling the question lies in the fact, as Clark pointed out, that the dispersion of aggregates cannot readily be distinguished from the disruption of crystal fragments by their end products.

Among fine-grained sediments in which few authigenic changes have taken place there should theoretically be little difficulty in effecting dispersion into the individual particles, but even here there is an increase of fine material with an increase of vigor or time in the dispersion process. If the sediment is indurated or altered by weathering, the original size distribution may have suffered considerable change due to secondary growth, dehydration, the introduction of secondary minerals, or the leaching out of certain constituents. The reconstruction of the original distribution may accordingly be nearly impossible. Clark's point thus applies in part to sediments as well, and the problem raised is not one that can be readily solved. It may be possible, however, to choose dispersion procedures which as far as possible meet the conditions set, with the realization that while the final picture is not completely accurate, it is at least more than a first approximation to the truth.

GENERALIZED DISPERSION ROUTINE

The entire subject of dispersion revolves about the point previously mentioned: the aggregates must be destroyed without affecting the sizes of the individual particles. There is a further condition implied in this process: the dispersed particles should not form aggregates again during the course of the analysis. To satisfy these conditions, shearing stresses and abrasive action on the individual particles should be kept at a minimum, while disaggregation and dispersion should be at a maximum. It seems to the writer that a series of physical methods of increasing vigor, combined with a peptizer, and the optional washing out of foreign electrolytes, should give the best results.

Gessner (7) devoted considerable

space to the dispersion of samples, and presented a general routine which involved several procedures, each followed by tests for dispersion and flocculation, so that the treatment given depends on the difficulty of dispersing the material. In his routine the sample is shaken and then tested for flocculation. The test shows either complete dispersion, incomplete dispersion without flocculation, or flocculation. The first case is analyzed, the second is boiled, the third is washed. The boiled sample is tested, and either analyzed or washed. The washed samples are either analyzed or rewashed.

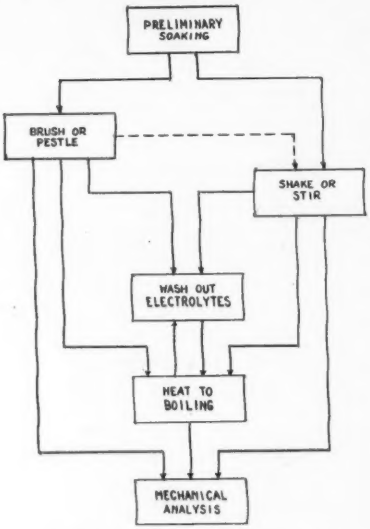


FIG. 2. Chart showing generalized dispersion routine.

A routine of this type appears to be the correct approach to the problem, inasmuch as it recognizes the need for different procedures in different cases. It also delays the washing until its necessity is indicated. The flocculation tests are important, since they afford a control which indicates the course of dispersion as the entire routine is followed.

As the result of a series of comparative tests made on certain of the dispersion procedures reviewed earlier, the writer developed a routine for dispersing fine-grained sediments in which the procedures become successively more vigorous, so that only the more resistant sediments receive the most vigorous treatment. Figure 2 shows the routine graphically. As in Gessner's plan the foreign electrolytes are washed out only when the need is indicated. In every case the sample is soaked in dilute peptizer for a preliminary period. This is followed by one of two sequences, depending on the amount of material above 0.06 mm. diameter in the sediment. By grinding a fragment between the teeth or rubbing between finger and thumb, the amount of sand present may be estimated. No fixed proportion is involved, but the writer has found that in most cases if only a trace of coarse material is present, brushing or pestling are more effective than shaking, while if there is an appreciable amount of sand, shaking or stirring are preferable. This is particularly true of unconsolidated sediments, but in partially indurated cases it is desirable to brush the sample before shaking it. The dashed line in the figure indicates this possibility. The several arrows illustrate the paths that may be followed. An attempt was made to allow some flexibility and yet to pass from gentle to more vigorous steps as the need was felt. In many

cases sediments are fully dispersed after brushing alone or shaking alone, but the more indurated shales studied required several steps. In only one case was washing necessary. It should be mentioned that vibration was not used because of lack of apparatus, and the absence of that procedure is not to be considered as excluding it from the routine.

The following description of the several procedures is arranged in the order of the two main sequences, but any steps common to both are described only once.

The air-dried[5] sediment is crushed with a rubber-tipped pestle, a rolling pin, or a wooden mallet until the fragments are reasonably small. A test sample is then quartered and weighed. The weight of the sample depends on the range of sizes present. It is desirable to have a suspension of 2 to 3 per cent concentration for analysis by modern methods, so that about 25 grams are optimum for a liter of suspension. If the sample contains say 25 per cent of sand, the test sample should weigh about 30 grams. When the sand is later sieved off, the remaining fine material will yield a suspension of about 2.3 per cent concentration.

The quartered sample is placed in a 250 cc. Erlenmeyer flask with 100 cc. of N/100 sodium oxalate solution[6] and allowed to soak for a period depending on the rate at which the lumps disaggregate. A minimum of 24 hours, and a maximum of 8 weeks, are possible limits to the time. Some Pennsylvanian shales required 10 days of soaking before they yielded to the brush, while others needed only 24 hours. By occasional shaking, the process of disintegration may be observed, and if the sample is taken out too early, it may be returned to the flask after brushing or pestling is found ineffective on the lumps.

After the preliminary soaking the sample is poured into an evaporating dish, the water is decanted and the paste is rubbed with a stiff brush or rubber pestle. The writer favors a brush for the purpose. As the lumps disaggregate, water is added and the dispersed material poured into a beaker. The water used during brushing should have a concentration of N/100 sodium oxalate. The brushing process often requires the better part of an hour, but the final results justify the use of ample time. Whittles (22) prepared a rubber pestle by filing down a stopper and attaching it to a glass rod. Such pestles are advantageous as an aid to brushing, because the more resistant lumps may be gently crushed before brushing.

The volume of suspension after brushing may be about 400 cc. It is tested for complete dispersion by placing a drop on a slide under a cover glass, allowing it to rest for a few minutes, and examining it under the microscope. If each grain stands out as an individual, and the smaller ones display Brownian movement, the dis-

[5] If it is preferred to work with sediments in their natural moist condition, the moisture content may be determined from a separate sample.

[6] It is convenient to have an N/5 solution of sodium oxalate on hand, prepared by dissolving 13.4 grams of the salt in a liter of water. The N/100 solution is made as needed by adding 5 cc. of this solution to 95 cc. of water.

persion is probably complete. If bead-like strings and clusters of individual particles extend through the field, the suspension is flocculated. If the field shows a mixture of individual grains and aggregates, not clustered together, flocculation is probably absent, but dispersion is incomplete. In practice there is considerable gradation among these three situations, and it is often difficult to distinguish among them. Further, if slow flocculation is present, the suspension may remain apparently dispersed for several hours. As a final test the writer allows the apparently fully dispersed suspension to stand overnight, and if visible flocculation has not set in within that time, it will not occur during the analysis. In very few cases suspensions have remained apparently dispersed for two or three days, and finally flocculated.

If the test shows complete dispersion, the suspension should be diluted to a liter with N/100 sodium oxalate, and analyzed. The writer favors the pipette method (11) for fine-grained sediments.

If dispersion is incomplete, the suspension is diluted to about 800 cc. with N/100 sodium oxalate, and heated to the boiling point. As soon as the liquid boils it is withdrawn from the flame. After cooling, the suspension is tested, and in every case studied by the writer, the samples were either fully dispersed or flocculation had set in.

If flocculation is present at any stage of the routine, the suspension, diluted to about a liter, is poured into a tall beaker and the liquid filtered through a Pasteur-Chamberland filter with suction. The set-up involves the filter connected to a filter bottle, with the outlet of the bottle connected to the filter pump. The suspension passes into the filter (which is a cylindrical tube) and collects in the bottle. When the residue in the beaker is like paste the filter is scraped and the sediment again made up to a liter with N/100 sodium oxalate, since most of the original peptizer has been withdrawn by the filtration. The suspension is again heated to boiling, and usually it will be dispersed. In some cases more than one washing is necessary. It is conceivable that some samples may be quite obstinate in their resistance to dispersion.

When the sediment has an appreciable amount of sand in it, the preliminary soaking is followed by shaking or stirring, unless the sediment is indurated, in which case brushing should precede the shaking or stirring. The sample is shaken for an hour, preferably in a reciprocating shaker, or it is stirred in an electric drink mixer for 5 or 10 minutes. It is important to use wire baffles in the cup (1). After the shaking or stirring, the suspension is tested for dispersion as described above.

If dispersion is incomplete, the suspension is diluted to about 800 cc. and heated to boiling, as described above. If coagulation is present, the foreign electrolytes are washed out as described above.

As in the writer's earlier routine, the material above 1/16 mm. diameter is sieved from the sample before analysis. The suspension is poured through the sieve and the liquid col-

lected in a beaker. The residue is washed with a gentle stream of water. The total volume of suspension should be less than a liter, including the wash water. The writer sieves the suspension after brushing or shaking, but it may be done at any convenient point in the routine. The material above 1/16 mm. is sieved dry into grades if it is appreciable in amount. It should also be examined to determine whether it consists of individual grains or undisintegrated aggregates.

Although no examples were found among those studied, in which this general routine did not produce dispersion, it is to be anticipated that cases will be met in which all efforts result in failure. In obstinate cases a long period of soaking should be followed, with brushing or pestling at intervals during the soaking. It is also desirable to determine the amount and nature of the foreign electrolytes, and to test the dispersive effects of other concentrations of sodium oxalate or sodium carbonate. A series of preliminary tests on small samples, such as may be made in test tubes, is often of value in indicating effective procedures.

CONCLUDING REMARKS

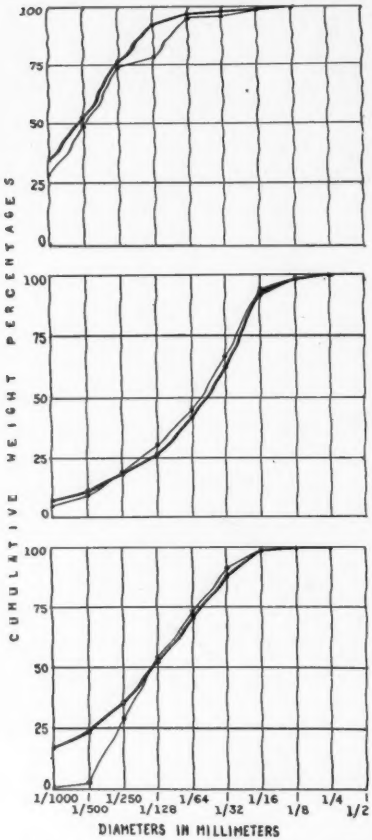
The writer's routine was tried on glacial tills, lake clays, loesses, and a number of Pennsylvanian shales ranging from soft gray shale to rather hard "soapstone" and black shale. Lack of space prevents the presentation of all the experiments, but three cases are cited, to show the effects of successive procedures and the results of flocculation. The three sediments represent a shale, an underclay, and a siltstone from Fulton County, Illinois. The analytical data are given in Table II.

Figure 3 shows duplicate analyses of Upper De Long (Pottsville) underclay. The unwashed sample was soaked in N/100 sodium oxalate for 8 days, then rubbed with a brush. The suspension was divided into two parts, one of which was heated to boiling. The boiled sample (heavy line) displays a smooth curve throughout its length, while the unboiled sample has a marked irregularity in it. The effect of boiling appears to have been the dispersion of aggregates in the irregular portion of the curve, and an increase of material below 1 micron. Heating to boiling has thus aided dispersion in this case.

TABLE II. *Analyses of three Pennsylvanian sediments*

Diameters in mm.	Upper De Long underclay		Purington shale		Babylon shale	
	Brushed	Brushed, boiled	Brushed	Brushed, boiled	Unwashed	Washed
Above 1/16	0.9	1.7	5.8	5.8	2.0	3.8
1/16–1/32	4.9	trace	28.0	33.0	8.0	10.4
1/32–1/64	0.0	0.8	22.7	21.2	16.8	15.6
1/64–1/128	16.2	5.0	13.5	14.5	18.5	17.1
1/128–1/250	4.0	15.0	13.5	10.0	28.7	15.3
1/250–1/500	23.4	26.5	7.0	4.4	24.1	14.4
1/500–1/1000	22.1	15.5	4.5	3.6	1.9	7.2
Under 1/1000	28.5	35.5	5.0	7.5	trace	16.2

Figure 4 shows duplicate analyses of Liverpool (Carbondale) siltstone. This sample only required 24 hours of soaking. It was brushed and one-half was heated to boiling. Here the boiling had little effect, and brushing alone yielded a fairly satisfactory analysis.



FIGS. 3, 4, AND 5. Analyses of three Pennsylvanian sediments. See text for explanation.

Figure 5 shows duplicate analyses of black Babylon (Pottsville) shale. This sample contains more than 1 per cent of water-soluble salts, among which ferric sulphate is prominent. An unwashed sample was soaked for 7 days, brushed, and heated to boiling. The boiled suspension flocculated, but an analysis (the light line) was run to determine the sizes affected. Another sample was soaked in distilled water for 36 hours and the soluble salts filtered off. The sediment was then soaked for 6 days, brushed and heated to boiling. In this case no flocculation occurred. The dispersed sample has 16.2 per cent of material below 1 micron, while in the flocculated suspension there was practically nothing below 2 microns. Beyond the 1/128 mm. ordinate, however, the curves parallel each other. Thus the sizes affected here are mainly under 8 microns in diameter.

These several cases represent the situations met in the other sediments studied, and indicate that while no single procedure effected dispersion in all cases, the choice of procedures offered in the routine was successful in every instance.

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PAINE, S. G., LINGGOOD, F. W., SCHIMMER, F., AND THRAPP, T. C. The Relationship of Micro-Organisms to the Decay of Stone; *Philos. Trans. Royal Society of London,* Ser. B, Vol. 222, pp. 97-127, 1933.

This paper gives the results of numerous experiments made to determine the influence of micro-organisms in the decay of building stone. The materials used in the experiments were derived from buildings in Great Britain and from British quarries. They include limestones, sandstones, slate, and granite, most being limestones and sandstones.

The authors of the paper sought answers to the questions as to whether bacteria exist in the interior of stone, whether they are more numerous in stone exposed to the weather, what depth they extend in stone, whether the species found in stone are adapted to the particular habitat, and whether the bacteria produce decay in stone.

It was found that many species of bacteria exist in building stone both in buildings and in quarries. The number per gram was found in the stone used in building to range from none to over two million in a stone from the north wing balustrade of Buckingham Palace, London. The authors caution that the figures they give are to be considered approximations and that the number per gram may be several times that given in the tables. The number per gram in quarry stone was found to range from none to nearly three million. It is concluded that the distribution of the bacteria in stone is very uneven and the figures indicate that the number per gram decreases with distance from the surface.

The bacteria were studied to determine the products produced by them and the effects of these products on rock decay and it was found that carbon dioxide is the most important product, but that there is some sulphuric acid and some nitrogen compounds.

The general results of the experiments are stated in the author's summary as follows: That bacteria are present in decaying stones of buildings and quarries: that the heterotrophic bacteria are related to soil, air, and water types, but have certain features that distinguish them as a group from the soil bacteria; that many of the heterotrophic bacteria produce acids by fermentation of organic compounds; that the nitrifying bacteria are probably chance infections from soil and not essential factors in rock decay; that sulphur-oxidizing bacteria may be isolated from decaying stone and may be important in rock decay; and that the sulphur bacteria grown in a culture medium are embedded in masses of "mucilage" and if they so grow in rocks it is possible that some disintegration in addition to decomposition may be produced through expansion and contraction of the "mucilage" due to changes in water content.

There are three plates illustrating seventeen examples of rock decay ascribed to the work of micro-organisms.

This contribution is considered important and suggests that micro-organisms may play a prominent role in rock decomposition.

W. H. TWENHOFEL

STAUFFER, CLINTON R., AND THIEL, GEORGE A., The Limestones and Marls of Minnesota. *Minnesota Geological Survey, Bulletin 23,* 1933. 193 pp., 93 figs.

That limestone, dolomite, and marl deserve this special attention is attested by the abundance of these rocks in Minnesota and the widespread requirement as a neutralizer of acid soils and in numerous other uses. The importance of calcium carbonate in a variety of industries is obvious after an inspection of the long list of uses given in the introduction of this useful report.

The report contains an eight page introduction and two principal parts. Part

I discusses the limestones and dolomites of the state under chapter headings of (1) The Nature and Origin of Limestone and Dolomite, 13 pages, and (2) Limestones and Dolomites in Individual Counties, 52 pages, and includes chemical analyses of 160 samples. Part II, by the junior author, is on the marls of Minnesota, discussed in four chapters; (1) The Nature and Origin of Marl, 13 pages; (2) The Relation of Marl Deposits to Different Types of Glacial Drift, 20 pages; (3) Prospecting for Marl, 4 pages; (4) Marl Deposits in Individual Counties, 81 pages, and includes many analyses of the marls showing per cent soluble carbonates.

The chapters on the nature and origin of the rocks studied are elementary but interesting. The authors state that limestones have been formed by organic and inorganic processes, and that "by far the majority of those limestones of which the history can be definitely determined are of organic origin." The reviewer doubts if this statement will hold for the limestones of the geologic column. In many of the lower Paleozoic limestones of the Upper Mississippi Valley the great bulk of the rock fails to show the structures of organic origin, but instead the matrix (and by far the greater bulk) of a fossiliferous limestone is composed of anhedral or euhedral grains of calcite or dolomite of varying size, depending on the texture of the rock. This granular texture is due to inorganic processes; it is immaterial if the calcium carbonate was derived from shell material before the inorganic precipitation.

Some of the common causes for the precipitation of calcium carbonate by inorganic processes are listed. The authors state that some of the oolites may have had an origin related to agitation of water near the saturation point for calcium carbonate. No important travertine deposits are known in Minnesota. The formation of limestones by purely mechanical processes is not considered.

Short paragraphs discuss the weathering and erosion of limestones, the characteristics of limestone regions, dolomitization, age and distribution, and the resources of limestone in the state. The authors conclude that the "dolomitization has been due largely to a replacement of part of the calcium . . . probably before the resulting rock had been completely consolidated." The limestones discussed belong chiefly to Cambrian, Ordovician, and Devonian systems. A statement is made concerning the various physical tests made on limestone to determine its fitness as a road metal.

The discussion of limestone in the counties containing outcrops is useful. Measured sections, physical tests and reference to the chemical analyses are included. Suggestions are made as to suitable uses, and attention is called to the desirability of dolomitic limestones in the treatment of acid soils. It appears that less of this type of rock is required per acre than of the high calcium limestone. A base map of the state showing counties and cities used in the location of these deposits would have added to the value of this chapter.

Part II on Marls contains much useful reference material. A good description of marl is given and the derivation of the calcium carbonate from the glacial drift is explained. In the discussion of causes of deposition is a good statement of the theories of chemical and biochemical precipitation and mechanical sedimentation. A classification of lake basins, the mode of occurrence of marl deposits, the nature of the material associated with the marl, are included in the first chapter. In Chapter II a full explanation is given of the relation of marl deposits to different types of glacial drift and includes physical and calcium carbonate analyses of the drift. It appears that the texture of the drift is more important than the composition in furnishing material for marl deposits, as the red, stony drift areas contain many more marl deposits than do the gray limy drift areas; the impervious character of the latter drift re-

tarded the leaching action. High morainic hills favor deposition of marl because of the good circulation of ground water, and partly due to an artesian condition common in these areas. Chapter III relates the methods used for prospecting and describes the Davis and Michigan types of samplers. A list of newly found deposits is included. Chapter IV describes the marl deposits of individual counties and contains the specific location, thickness, character and extent of each known deposit.

In addition to the direct value of this publication to the people of Minnesota it has much of use to the students of stratigraphy and sedimentation.

A. C. TESTER

PIA, JULIUS. *Die Rezenten Kalkstein, Zeitschrift für Kristallographie, Mineralogie und Petrographie,* Abteilung B, 1933, pp. 1-420, 4 plates.

This important work by Pia has an introduction of 18 pages in which are considered the classification of the calcareous sediments, and the solubility of calcium carbonate. Chapter I treats of the limestones due to inorganic causes or processes and these so developed are designated abiogenic limestones. In Chapters II and III there are considered the limestones that are due to organic agencies, as travertine and evaporation deposits. These are divided into the physiologenic limestones or those arising from the vital activities of organisms, as algal crusts, and the organic limestones composed of organic remains which were parts of the organisms, as the shells and corals. Each of these in turn is subdivided.

This monographic work on the recent calcium carbonate sediments is easily the best work on the subject that so far has been published. It should be on the desk of every sedimentationist who is interested in this group of sediments.

W. H. TWENHOFEL

HADDING, ASSAR. *The Pre-Quaternary Sedimentary Rocks of Sweden, Part V, On the Organic Remains of the Limestones.* Lunds Universitets Arsskrift, N.F. Avd. 2, Bd 29, Nr. 4, 1933, Lund Sweden, 93 pp., 53 figs.

This is another contribution by the same author to a thorough study of the pre-Quaternary sedimentary rocks of Sweden. In this publication of 93 pages the limestone forming organisms are described in systematic order. The plants include only the bacteria and algae; the animals are divided into the usual phyla of invertebrates, and the vertebrates are treated under one heading.

The author is cautious to distinguish the use made of fossils by the sediment-petrographer and by the paleontologist or biologist. The stratigraphic significance of fossils does not coincide with the petrographic value; their latter value is partly because they are rock forming and partly because they inform the petrographer of the environmental conditions under which the rock was formed. However, this very difference in value is of considerable interest. Heretofore the organic remains have received the attention of paleontologists as documents of time; now, the sediment-petrographer studies the fossils as documents of milieu. The study of organic remains by Hadding is made on this basis. Throughout he attempts to determine the milieu as indicated by each class of organism.

The space of this review does not permit a citation from each organic group. The discussion of algae as limestone formers is noteworthy. Precipitation of calcium carbonate results from the extraction of carbon dioxide from the water by algae, and as in the case of bacteria can take place quite independent of these organisms' own structure. The marine plankton Coccolithophoridae are considered to be important and of considerable petrographic interest. Coccoliths are present in a number of the rocks of Cretaceous age. Because of the minute size of the algae and the transformation of the older rocks these forms have not been recognized in the Paleozoic limestones. However, in recent deposits coc-

coliths have been found in most kinds of marine sediments except the detritus deposits along the shores; they occur in small numbers in the red deep-sea clay at a depth of more than 6,000 meters and are most abundant in the globigerina ooze. Coccoliths are abundant in chalk and may be of considerable significance in its formation.

In a number of places the author emphasizes the slight depth of the marine waters in which the limestone forming organisms lived. Especially is this true of a number of algae, corals, and ostracodes. A caution is expressed concerning the interpretation of milieu from fossils, especially certain mollusks, as they could have lived, died, and been buried in quite different places.

In summary, the author stresses the point that "a group of animals or plants does not alone form the material of which a limestone is built up" and he repeatedly points out "that the one or the other fossil group is not so abundantly present in the rocks as to form the bulk of them."

In parts of the series to be published later the author will give an account of the petrographic analysis of different types of limestone, and will discuss the environmental conditions under which the different fossils have developed.

A. C. TESTER

MOHR, E. R. J. *Tropical Soil forming Processes and the Development of Tropical Soils, with Special Reference to Java and Sumatra.* Translated from the Dutch by R. L. Pendleton, *National Geological Survey of China,* 1933, pp. 200.

This work on tropical soils, translated from the second edition (1930) of *De Grond van Java en Sumatra,* is of much interest to sedimentationists as it describes the early stages in the production of sediments under the conditions of a tropical climate. Much has been written on soil formation under the conditions existing in north temperate latitudes and the information with respect to the soils of temperate regions as immediate sources of sediments is extensive, but knowledge of tropical soils as sources of sediments is limited. The reviewer is grateful to Professor Pendleton for undertaking the translation of Dr. Mohr's work and to the National Geological Survey of China for publishing the translation.

The book is divided into two parts. In Part 1 general principles are considered. Part 2 describes the local conditions with respect to soil formation in different parts of Java and Sumatra. Part 1 treats of the soil-forming rocks of Java, the climates of Java, the forms of weathering in that region, the stages of weathering, the autochthonous soils to be expected, and the secondary (allochthonous) soils. These six chapters cover 52 pages.

The soil-forming rocks, with minor exceptions, are eruptives and Tertiary limestones, shales, sandstones, and conglomerates. The weathering is stated to be almost exclusively chemical, that is, decomposition. The soils produced result from the various combinations of temperature, rainfall, evaporation, and relief, the last not distinctly stated. He distinguishes seven types.

1. High temperature with rainfall always higher than evaporation produces laterite or laterite soils. This type of soil is designated lixivium, meaning a thoroughly leached product.

2. High temperature and rainfall with more or less dry season produce a silicic-acid-poor lixivium of red color.

3. Low temperature with rainfall continuously higher than evaporation produces a lixivium in which the silicic acid is retained.

4. High temperature and rainfall with a dry period produce dark soils containing much organic matter and also containing concretions.

5. High temperature with rainfall continuously less than evaporation leads to the formation of saline soils of brownish-gray color.

6. High temperature with rainfall continuously higher than evaporation pro-

duces with under-water weathering light-colored soils composed of little else than kaolin and silicic acid.

7. High temperature and climate with a rainy season and a dry season produces black soils under conditions of partial submergence.

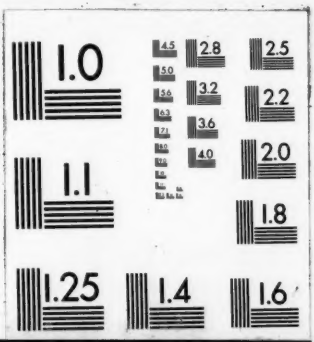
There is given a table showing the soils resulting from the different combinations of the conditions.

Chapter V discusses the forms of soil developed in Java and Sumatra from the different rocks present there and under the conditions of the various combinations of rainfall, evaporation and temperature. Chapter VI describes allochthonous or transported soils; river deposits, marine deposits, and wind deposits being considered.

The second part of the work, covering 148 pages, is concerned with local conditions of soil development and is of great interest. It contains much of importance to the sedimentationist. Because of the detail it does not readily lend itself to summary review.

This work on tropical soil formation should be read by students of sediments. Soils are the immediate parents of most of the shales and sandstones and also of some of the gravels and materials deposited from solution. Knowledge of soils is essential to the sedimentationist and intelligent interpretation of a sedimentary terrane consisting of shales or sandstones involves the parent from which the materials were derived and this parent in turn predicates a certain combination of temperature, rainfall, evaporation and relief.

W. H. TWENHOFEL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